A952065 (1)

DEPARTMENT OF THE NAVY
OFFICE OF NAVAL RESEARCH
(ADVANCED RESEARCH PROJECT AGENCY)

Contract Nonr 1858(32) - NR 098-201

BURNING RATE CONTROL FACTORS IN SOLID PROPELLANTS

Fourth Quarterly Status Report

For the Period 1 October 1959 to 31 December 1959

Aeronautical Engineering Report No. 446 d

Prepared by: Kimball P. Hall
Research Associate

and E. Karl Bastress
Research Assistant

Approved by: Kimball P. Hall
for Martin Summerfield
Principal Investigator

28 March 1960

Department of Aeronautical Engineering
PRINCETON UNIVERSITY
Princeton, New Jersey

TABLE OF CONTENTS

I. INTRODUCTION

The status of the research under this contract has been described in a recently published Technical Report (Reference). Topics covered in Reference 1 include: (a) strand burner burning rates techniques, (b) burning rate measurement in rocket motors, (c) effect of oxidizer particle size on burning rate, and (d) role of radiation as a mechanism of energy feedback in the composite propellant combustion process.*

Since the Technical Report provides adequate coverage of most of our research activities under this contract through 31 December 1959, the present Status Report will cover only one research topic; namely, the development of techniques for particle size analysis of ground ammonium perchlorate oxidizer, using the Mine Safety Appliances Particle Size Analyzer.

During this quarter Princeton University and the Office of Naval Research concluded the contractual arrangements authorizing the University to initiate construction of the badly needed new Solid Propellant Processing Building.

II. PARTICLE SIZE ANALYSIS USING MSA ANALYZER

It is customary in the technology of present-day ammonium perchlorate composite solid propellants to incorporate into the propellant mix both coarse (unground) and fine (ground) oxidizer. The particle size distribution in the unground material can readily be evaluated by screen analysis, but the determination of the distribution in ground material, (which is finer than the

---

*Topic (d) is based on research partially funded by Project SQUID.

finest screens commonly available) requires the use of much more elaborate and sophisticated techniques. In the solid propellant industry, this analysis is normally performed by means of an air sedimentation device known as the Micromerograph.* We have recently acquired a different device for this purpose, and have evaluated its performance relative to the Micromerograph, with impressive results.

The device referred to, manufactured by the Mine Safety Appliances Company (Pittsburgh), is the MSA Particle Size Analyzer. (For photographs of the apparatus see Reference 1, Figures 8 and 9.) As in the Micromerograph, size distribution is evaluated on the basis of sedimentation time (assuming spherical particles); but the sedimentation fluid is a liquid instead of air.

Although the Mine Safety Appliances Company supplies a manual of operating procedures for the MSA Analyzer (Reference 2), it was necessary for us to devote a considerable effort to the development of techniques for analyzing ammonium perchlorate. One of the more difficult problems was the selection of suitable fluids for dispersion and sedimentation.** The procedure and our modifications of it will, therefore, be outlined briefly.

A sample of the material to be analyzed is allowed to settle in a special centrifuge tube, the lower portion of which is a precision bore capillary. By means of a magnifying projector the height of the column of material that has accumulated in the capillary is read at intervals, in accordance with a precomputed time schedule, such that the instant at which each reading is taken corresponds to a particular particle size.

---

*Manufactured by the Sharples Corporation, Philadelphia, Pa.

**We were unable to obtain results with the fluids recommended by MSA for ammonium perchlorate; namely, two commercially supplied saturated hydrocarbon "cuts".

After those particles which settle in a reasonable time under gravity have settled out, the sample is centrifuged to shorten the settling time of the finer particles. The final product is a table correlating column height with Stokes equivalent particle diameter.

By assuming that

$$\frac{\Delta h}{h_{total}} \times 100 = \text{weight percent}$$

(where h = height of column in capillary

Δh = increment of height between two sizes)

the tabulated results may be used to plot a particle size distribution curve. Our tests of the validity of the above assumption are reported in the next section.

In addition to performing validity checks, we conducted a search for liquids which would be suitable for ammonium perchlorate analysis, and investigated methods for effectively dispersing the sample without introducing errors into the procedure. Some experimentation was also necessary to determine how best to incorporate the centrifuging steps into the settling schedule for greatest precision.

The analysis procedure requires the use of a dispersion fluid in which the sample is first suspended, and a slightly more dense sedimentation fluid in which the settling actually occurs. The operating steps are outlined in Figure 1. The fluids and dispersion techniques which we finally selected depended on the nature of the sample, as shown in Table I. For a typical fine sample our procedure calls for gravity settling of particles greater than 20 μ (time required at 75°F, 412 seconds), centrifuge settling at 300 RPM of all coarser than 10 μ (total time of centrifuging 141 seconds), centrifuge settling at 600 RPM of all coarser than 5 μ (total time of centrifuging

176 seconds), and centrifuge settling at 1200 RPM of finer particles (114 seconds for all above 3μ, 22 minutes additional for all above 1μ). The total run time for a sample is less than twenty minutes (unless sizes below 3μ are to be analyzed).

III. TESTS OF THE METHOD'S VALIDITY

These tests took two forms: first, attempts to determine whether increments in column height are equivalent to increments in weight fraction of the sample; and second, comparison of analysis results obtained at Princeton by this method to analysis results obtained by two other organizations using different methods but operating on the same samples of ammonium perchlorate.*

The results of the comparative analyses are summarized graphically in Figure 2.

The assumption of equivalence of volume fraction (column height fraction) to weight fraction was shown to be valid in the following way. A sample of ground ammonium perchlorate was dispersed in benzene with a Brookfield stirrer. Portions of the suspension were inserted into six different sedimentation tubes. Settling was allowed to proceed according to the appropriate schedule until all particles greater than a given size (different for each tube) had settled (using the appropriate centrifuges where called for).

The height of the settled column was then read, and the fluid suspension above the column was immediately removed. The solvent remaining

---

*The samples tested contained anticaking agent. They were supplied by the courtesy of Dr. Henry Shuey of the Redstone Arsenal Research Division, the Rohm and Haas Company. They were first analyzed at Rohm and Haas, later analyzed by us, and finally analyzed by the Elkton Division of the Thiokol Corporation, through the courtesy of Dr. Gordon R. Leader.

in the six tubes was driven off by oven drying, and the tubes were weighed on an analytical balance. The weight of the column of oxidizer* in each tube was determined by difference, and the weight per unit of column height in each tube was computed. The value, weight per unit height, was the same for all six tubes within a spread of $\pm$ 2%. (See Table II.)

*The measured weight ranged from 4 milligrams for the shortest column to 15 milligrams for the longest.

REFERENCES

1. Blair, Bastress, Hermance, Hall, Summerfield, "Some Research Problems In the Steady-State Burning of Composite Propellants," Aeronautical Engineering Report No.499, Princeton University, 10 March 1960. (Technical content identical to American Rocket Society Preprint No. 1060-60, same authors, same title.)

2. "M-S-A Particle Size Analyzer, Operating Procedures and Applications," Mine Safety Appliances Company, Pittsburgh, Pa.

TABLE I

FLUIDS AND DISPERSION TECHNIQUES

Sample Type	Dispersion* Fluid	Sedimentation Fluid	Method of Dispersing Sample	Centrifuge for Particles Below:
Fine (contains substantial percentage of particles finer than 10 microns)	benzene	chlorobenzene (viscosity about 0.7 cp.)	Brookfield counter-rotating stirrer; (1 gm. sample in 100 gm benzene)	20
Medium (10 to 100 microns)	benzene	chlorobenzene	by shaking in feeding chamber (20 mg sample)	20
Coarse (over 100 microns)	dibutyl sebacate	diethyl phthalate (viscosity about 10 cp.)	by shaking in feeding chamber	80

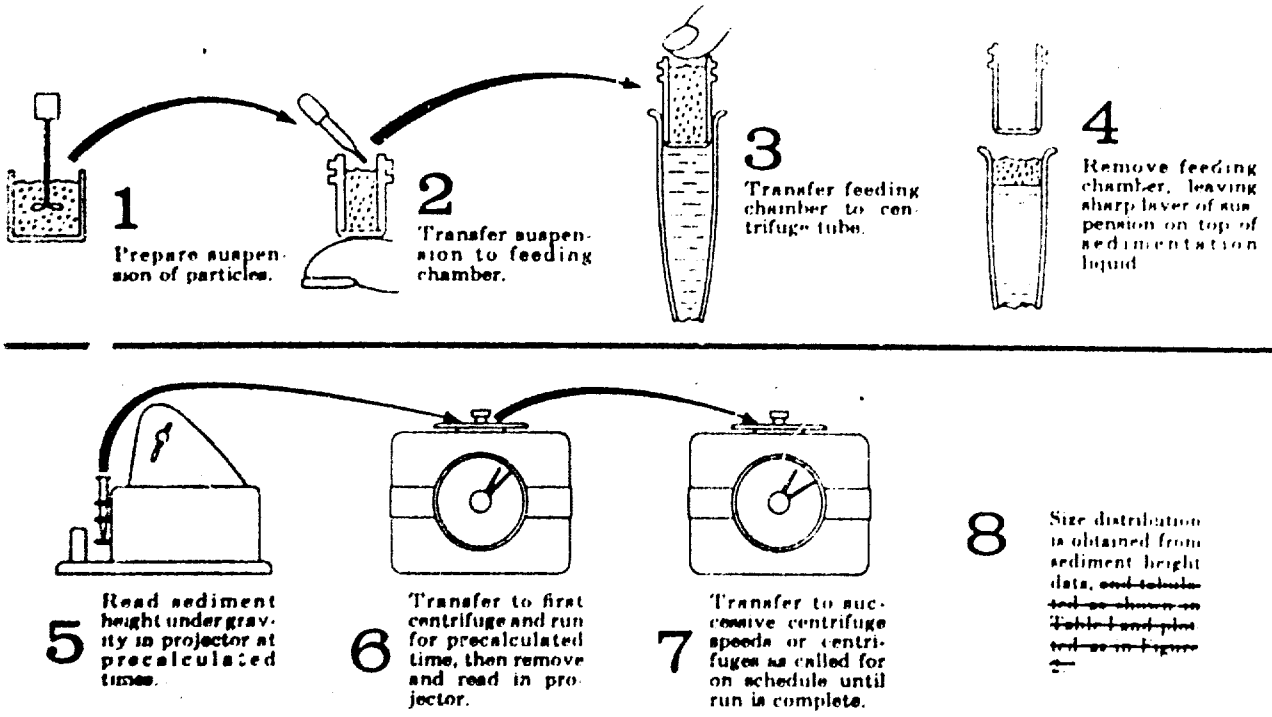
*0.05% of Twitchell #8240 dispersing agent (suggested by MSA) is added in each case.

TABLE II

TEST OF EQUIVALENCE OF VOLUME
FRACTION TO WEIGHT FRACTION

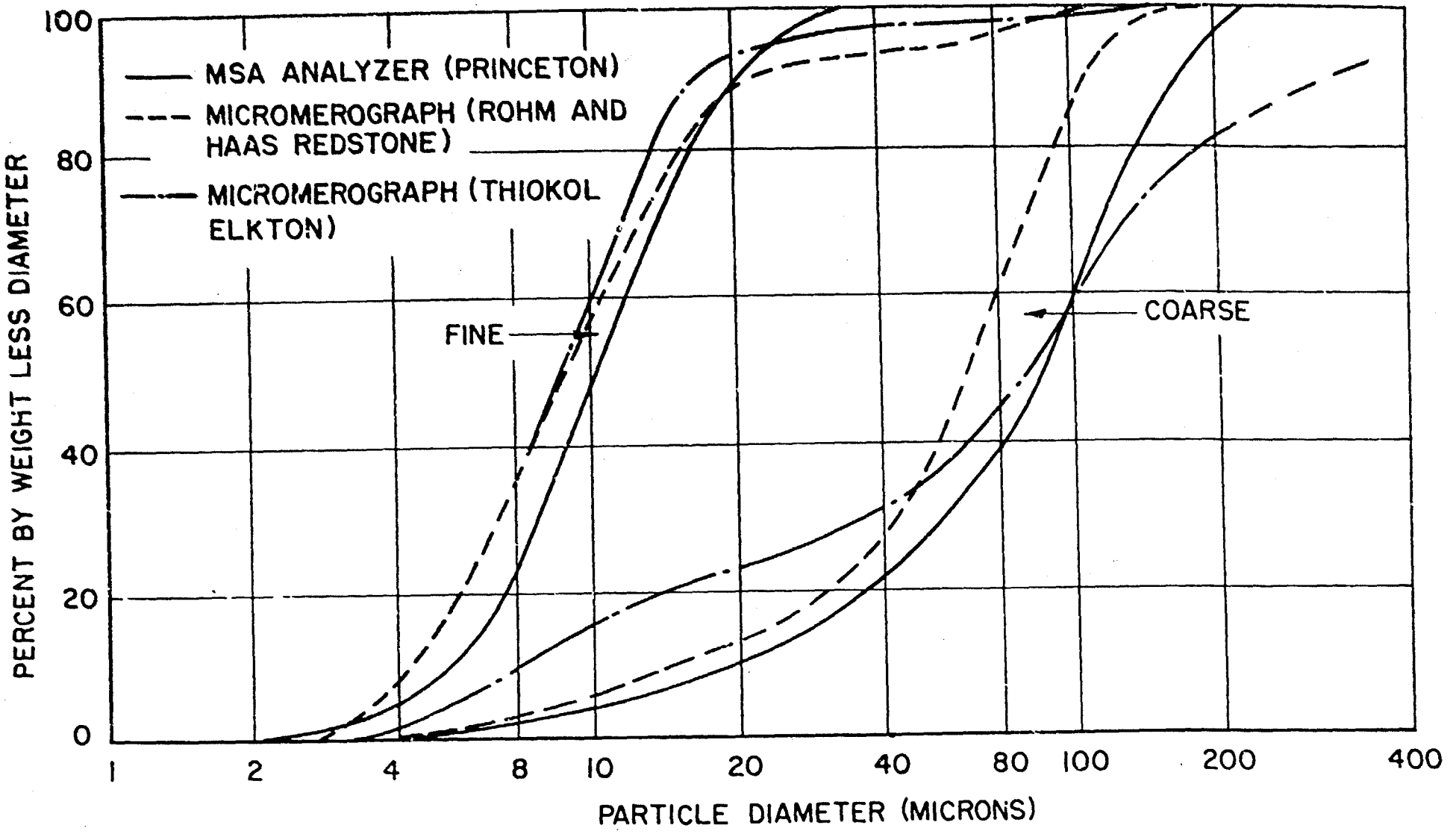
Sample Tube Number	Contains All Particles Greater Than	Column Height / Column Weight
1	60 microns	3.73 div./mg.
2	40 microns	3.75 div./mg.
3	25 microns	3.75 div./mg.
4	15 microns	3.60 div./mg.
5	7.5 microns	3.64 div./mg.
6	1 microns	3.65 div./mg.

Basic steps in particle analysis with MSA Particle Size Analyzer



(TAKEN FROM REFERENCE 2)

FIGURE 1



COMPARATIVE PARTICLE SIZE ANALYSES
GROUND AMMONIUM PERCHLORATE—TWO GRINDS

DISTRIBUTION LIST FOR STATUS REPORTS

Contract Nonr 1858(32)
NR 098-201

Agency	No. of Copies
Chief of Naval Research Department of the Navy Washington 25, D.C. Attn: Code 429	6
Chief of Naval Research Department of the Navy Washington 25, D.C. Attn: Code 426	1
Bureau of Naval Weapons Munitions Building Washington 25, D.C.	2
Office of Naval Research Chemical Science Building Forrestal Research Center Princeton, New Jersey Attn: Julian Levy Resident Representative	1